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Navy Case No. 77,897

Commissioner of Patents and Trademarks
Washington, D. C. 20231

Applicant: Imam et al.

For: "POROUS METAL/ORGANIC POLYMERIC COMPOSITES"

Sir:

Transmitted herewith are the papers above-identified
constituting a Patent Application filed by the Department of
the Navy on behalf of the above-named Applicant(s).

The total filing fee has been computed in accordance with
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Basic Fee -----\$ 770.00

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Respectfully,

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Enclosures:

(1) Application Papers

Navy Case No. 77,897

APPLICATION FOR LETTERS PATENT

TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN THAT M. Ashraf Imam, Bhakta B. Rath and Teddy M. Keller are citizens of the United States of America, and residents of Great Falls, Va., Oakton, Va., and Alexandria, VA., have invented certain new and useful improvements in "POROUS METAL/ORGANIC POLYMERIC COMPOSITES" of which the following is a specification:

Prepared by:
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Docket No.: N.C. 77,897
Inventor's Name: Imam et al.

PATENT APPLICATION

ORIGINAL

POROUS METAL/ORGANIC POLYMERIC COMPOSITES

Background of the Invention

1. Field of the Invention

The present invention relates generally to metal composites and more particularly to metal/polymer composites.

2. Description of the Background Art

In many cases, such as in factories, motor vehicles, ships, and submarines, it is desirable to absorb as much noise as possible. In factories, absorbance of machinery noises can minimize noise pollution caused by industrial sounds. In motor vehicles, absorbance of engine noises and noises from road vibration enhances driver and rider comfort. In submarines and ships, the absorbance of machinery sounds helps prevent detection and identification by unfriendly forces. Additionally, in regions where earthquakes are common, the structural components of buildings, bridges, and similar artifacts must not only be strong, but should be able to dampen vibrations.

Past noise reduction efforts have previously taken one of two approaches. The use of soft coupling components (e.g., bushings, pads) to isolate machinery, and the incorporation of structural acoustic damping materials.

1 Both of these prior art approaches have had drawbacks. Soft
2 coupling components add weight to a device, may require
3 maintenance, and may not be consistent with the required design and
4 performance features. Currently available acoustic damping
5 materials do not exhibit adequate acoustic behavior over broad
6 ranges of temperature, lack the required strain amplitude and
7 frequency, or do not have the desired strength and environmental
8 resistance.

9 Significant advances have been made to increase the strength
10 and stiffness of lightweight materials. Examples of these
11 developments include the superior density-normalized strength and
12 stiffness of Al-Li, hard dispersion strengthened aluminum and
13 titanium, aluminides, and whisker or fiber reinforced aluminum and
14 magnesium. Although those materials have excellent structural
15 characteristics, the demand continues for high strength,
16 lightweight structural materials that also vastly improve damping
17 capability.

18 United States Patent No. 4,759,000, the entirety of which is
19 incorporated herein by reference for all purposes, to Ronald P.
20 Reitz describes acoustically transparent windows made of an
21 aluminum/nickel foam impregnated with an acoustically non-absorbent
22 rubber such as BE silicone rubber RTV-11. Nothing in that patent
23 teaches or suggests that a useful material may be obtained by
24 impregnating a metal foam with an acoustically absorptive polymer.
25

Summary of the Invention

Accordingly, it is an object of this invention to produce a light structural materials having good structural and acoustic damping properties.

It is another object of the present invention to reduce noise derived from the operation of machinery.

It is a further object of the present invention to acoustically isolate internal areas of a structure from their surrounding environment.

These and other objects are achieved by impregnating a metal foam with a polymer.

Brief Description of the Drawings

A more complete appreciation of the invention will be readily obtained by reference to the following Description of the Preferred Embodiments and the accompanying drawings in which like numerals in different figures represent the same structures or elements, wherein:

Fig. 1 shows the acoustic damping behavior of an aluminum alloy foam/phthalonitrile composite, heat treated under the various condition described in the accompanying Example 3.

Fig. 2, Fig. 3 and Fig. 4 show the results of damping

1 measurements for a copper foam/phthalonitrile composite, over a
2 frequency range of 0.1 to 10 Hz.
3

4 Fig. 5, Fig. 6, and Fig. 7 show the damping behavior of a
5 titanium foam/phthalonitrile composite under different heat
6 treatment conditions as described in Example 5.
7

8 Fig. 8 and Fig. 9 show the damping behavior of a zinc
9 foam/phthalonitrile composite under different heat treatment
10 conditions as described in Example 5.
11

12 Fig. 10 is a graph showing the $\tan \delta$ over a frequency range of
13 0.1 to 10 Hz for various polymers and metal foams.
14

15 Fig. 11 shows the damping behavior of a white rubber/aluminum
16 alloy foam over the frequency range of 0.1 to 10 Hz.
17

18 Fig. 12 shows the damping behavior of a red rubber/aluminum
19 alloy foam over the frequency range of 0.1 to 10 Hz.
20

21 Fig. 13 shows the damping behavior of an epoxy/aluminum alloy
22 foam over the frequency range of 0.1 to 10 Hz.
23

24 Fig. 14 shows the damping behavior of an acrylic
25 polymer/aluminum alloy foam over the frequency range of 0.1 to 10

Hz.

Fig. 15 shows the stress/strain diagram for an aluminum alloy foam/phthalonitrile composite that was heat treated at 280°C for 18 hours, at 325°C for 4 hours, and at 375°C for 4 hours.

Fig. 16 shows the stress/strain diagram for an aluminum alloy foam/acrylic composite.

Description of the Preferred Embodiments

The metal foam of the present invention may be any porous metal workpiece, particularly a metal foam, regardless of shape or percent density, having an open cell structure that permits impregnation with an uncured polymer. Particularly useful metals include aluminum, titanium, nickel, copper, iron, zinc, lead, silver, gold, platinum, tantalum, and alloys (including steel) based on these metals. Other metals may also be used. Aluminum and titanium and alloys thereof are particularly useful because of their low density.

Metal foams may be produced by a variety of known methods. For example, a molten metal may be placed under high pressure so that it dissolves a non-reactive gas. When the pressure is released and the metal is allowed to cool, the dissolved gas escapes and leaves open-celled pores in the metal body. In other

1 processes, foaming agents are added to molten metal. Another
2 process mixes monomers with metal particles and heats the mixture
3 to induce foaming and solidification of the resin. As used herein,
4 the term "resin" encompasses prepolymers, monomers, and mixtures
5 thereof. A "cured resin" is the cured polymer. With further
6 heating, the metal particles consolidate and the polymer
7 hydrolyzes. United States Patent No. 4,569,821, the entirety of
8 which is incorporated herein by reference for all purposes,
9 improves upon that process by substituting a stabilized hydrogel
10 for the monomers, allowing more complete pyrolysis of the organic
11 components during formation of the metal foam.

12 In a foam, pore size is defined as the number of pores per
13 linear unit length. Because foams have only thin ligaments between
14 the pores, the void diameter of a pore is approximately the
15 reciprocal of the pore size of the foam. Decreasing the void
16 diameter increases the contact surface area between the metal foam
17 and the polymer. Also, as the percent foam density ($\% \text{Foam Density}$
18 $= \text{Density of a One Unit Volume of Foam} / \text{Density of One Unit Volume}$
19 $\text{of Metal} \times 100$) of the metallic foam decreases, the final product
20 increasingly exhibits the structural characteristics of the polymer
21 material. Thus, as the percent foam density of the metallic foam
22 decreases, the stiffness of the composite decreases. Even with
23 extremely low percent foam densities, however, the stiffness of the
24 composite is greater superior to that of either of the individual
25 components. Percent foam density and pore size also determine the

1 number of polymer/metal interfaces that an acoustic vibration must
2 traverse. As explained below, the polymer/metal interfaces
3 contribute mainly to the sound damping capabilities of the
4 composites of the present invention. A typical useful pore size is
5 5-100 pores/inch. More often, a pore size of about 10-50 pores/inch
6 is used, and most often a pore size of about 10-40 pores/inch is
7 used. Typically, the percent foam density is about 5-40. More
8 often, the percent foam density is about 8-10.

9 Typically, to improve the predictability of the structural and
10 acoustic properties of the composite products of the present
11 invention, the pores (also referred to in the present specification
12 and claims as "cells") within the metal foam have a locally uniform
13 size and distribution. Throughout the present specification and
14 claims, a foam has a locally uniform size and distribution of pores
15 if most of the pores of the foam are surrounded by evenly
16 distributed pores having approximately the same void diameter as
17 the surrounded pore. Local non-uniformity in the size or
18 distribution of the pores within the metal foam decreases the
19 ability to predict the characteristics of the resulting composite.
20 If desired, the metal foam may be divided into regions of different
21 pore size, or may have a gradation of pores sizes in any direction
22 along the metal foam, while maintaining locally uniform pore size,
23 without harming the predictability of performance.

24 The metal foam may be impregnated by any available method.
25 Typically, the metal foam is impregnated by contacting it with a

1 resin component. The resin component may be a neat resin or a neat
2 blend of resins, or may include any catalysts, curing agents, or
3 additives desired. The resin component may be a powder (of
4 sufficiently small particle size to penetrate the pores of the
5 metal foam), a melt, a room temperature liquid, or a solution, and
6 may include mixtures of several prepolymers and/or monomers. A
7 vacuum or positive pressure may be applied to assist the
8 penetration of the resin component into the metal foam. Solvent,
9 if present, is removed by evaporation. The resin component is then
10 converted (solidified or consolidated by any method, typically
11 heating to polymerize and/or cure the resin, or cooling to solidify
12 a molten resin) to a solid bulk polymer (partially crosslinked,
13 fully crosslinked, or non-crosslinked) that fills or partially
14 fills the open-cells of the metal foam.

15 The viscosity of the resin component (i.e., the impregnant)
16 can be adjusted both thermally or by adjusting the amount of any
17 solvent used, if necessary. Preferably, the impregnant viscosity
18 should be selected to allow the metal foam to be completely
19 impregnated with the resin component under practical processing
20 conditions. A high impregnant viscosity may restrict the ability
21 of the resin component to completely penetrate the open porous
22 structure of the metal foam. This problem may be overcome by
23 forcing, under positive pressure, the resin component into the
24 pores of the foam. For any given impregnant used, an appropriate
25 impregnant viscosity may be selected empirically, without undue

1 experimentation, given the guidance provided by this specification
2 and the accompanying examples. Resin component in powder form can
3 be forced into the pores of the metal foam by any method. For
4 example, the powdered resin component may be poured on top of the
5 metal foam, and positive or negative pressure may be applied to the
6 powder, forcing it into the pores of the foam. At the same time,
7 the metal foam may be vibrated to aid in impregnation.

8 The polymeric component of the present invention is typically
9 selected to have high intrinsic acoustic damping. Basically, the
10 acoustic damping ability of a polymer is determined by its dynamic
11 modulus at a given frequency. In the case of rubber materials, the
12 acoustic damping ability of unhardened rubbers is significantly
13 greater than that of hardened or fully hardened rubbers. In
14 general, thermoplastics and thermosets, unlike elastomers, provide
15 excellent results when used according to the presently claimed
16 invention, whether cured or uncured.

17 Polymers particularly useful in the present invention include
18 phthalonitriles, epoxies, acrylics, silicones, polyurethanes,
19 polyimides, polyvinyls, polycarbonates, natural rubbers, synthetic
20 rubbers, phenolics, polyolefins, polyamides, polyesters,
21 fluoropolymers, poly(phenylene ether ketones), poly(phenylene ether
22 sulfones), poly(phenylene sulfides) and melamine-formaldehyde
23 resins.

24 The acoustic damping capabilities of the composite of the
25 present invention arise in part from the acoustic properties of the

1 polymerized resin component and in part from dissipation of energy
2 at the polymer/metal interface. Energy is never transferred
3 without loss at interfaces between different materials. Therefore,
4 as the number of interfaces that an acoustic vibration must
5 traverse increases, the percentage of dissipated acoustic energy
6 also increases. A metal foam/polymer composite provides numerous
7 interfaces between the polymer and the metal matrix.

8 Additionally, relative motion between the polymer and metal at
9 their interfaces increases the loss of acoustic energy at these
10 interfaces. Thus, the acoustic damping properties of a
11 polymer/metal matrix composite may be improved, in some cases, by
12 forming the composite under conditions that avoid or minimize
13 chemical or bonding between the metal and the polymer component.
14 Bonding between the polymeric component and the interface may be
15 controlled by the combination of metal and polymeric agent
16 selected, the choice of curing agent and/or curing mechanism, and
17 the application of a release agent, if any, to the metal foam
18 before impregnation with resin component. In other cases, however,
19 vibration of a bond between a metal foam and a polymer may be a
20 loss mechanism that increases acoustical damping across the desired
21 frequency range.

22 For acoustic damping applications, the composite of the
23 present invention should provide a sufficient number of
24 polymer/metal interfaces to allow good acoustic damping. Thus,
25 particularly when formed as a sheet for acoustic damping

1 applications, the smallest dimension of the metal foam (for a
2 sheet, the thickness) is usually at least about 1.5 times the
3 average void diameter of the metal foam. In many cases, for
4 acoustic damping applications, the smallest dimension of the metal
5 foam will be three or more times greater than the average void
6 diameter of the metal foam.

7 Also, acoustic damping in these materials might be further
8 improved by the inclusion of polymer/gas and/or gas/metal
9 interfaces, which further increase the number of interfaces that an
10 acoustic vibration must traverse through the composite. These
11 polymer/gas and/or gas/metal interfaces may be formed by many
12 mechanisms, such as the use of a foamed resin component to produce
13 the polymeric component of the composite, the dissolution of the
14 neat resin or blend of resins in a solvent before impregnation, or
15 the inclusion of minute amounts of gas, or materials that form a
16 gas or a vapor during curing, within the resin component used to
17 form the polymeric component. The amount of gas or gas forming
18 materials should be sufficient to significantly enhance the
19 acoustic damping capabilities of the composite, but should not
20 provide sufficient gas to essentially destroy the structural
21 integrity of the composite and/or the polymeric component thereof.
22 The optimum amount of any gas or gas producing agent used in the
23 resin component will vary depending upon the desired use for the
24 composite and may be empirically determined without undue
25 experimentation.

1 The interaction of the metal foam and the polymer matrix also
2 contributes to the structural strength of the composite.
3 Consequently, the structural strength of the metal foam/polymer
4 composite is greater than the individual structural strengths of
5 the metal foam and the polymer.

6 Composites according to the present invention may be
7 constructed to exhibit acoustic damping across a frequency band
8 residing within a range of typically about 0.001-80 kHz. The
9 precise frequency band, as well as the bandwidth over which a
10 composite according to the present invention exhibits acoustic
11 damping, is determined by the selected polymer, metal, pore size
12 and percent foam density. The frequency range of damping may be
13 extending by stacking together metal foam/polymer composites having
14 different pore sizes, percent metal foam densities, polymers and/or
15 metals. Several sheets may be laminated together with adhesive, or
16 in some cases will bond to each other during curing of one or more
17 of the polymers within the metal foams, to form a laminate. Pore
18 size and percent metal foam density may also be varied, without
19 stacking, by providing a metal foam having regions of different
20 average pore size or having an average pore size that is graded in
21 one or more directions. It may also be possible to vary the
22 polymer used within a single sheet. For example, a foam may be
23 impregnated with a first resin component. The resin component-
24 impregnated foam may then be subjected to pressure on, for example
25 its upper surface, to force any portion of the first resin

1 component out in the upper portion of the metal foam down into the
2 lower portion. The resin component-impregnated foam may then be
3 solidified while under this positive pressure. After partial or
4 complete consolidation or solidification of the first resin
5 component in the lower portion of the metal foam, the upper portion
6 of the metal foam may be impregnated with a second resin component.

7 Aside from enhanced performance, components manufactured using
8 composites according to the present invention may alter the noise
9 properties of machinery, aerospace vehicles, domestic vehicles,
10 military vehicles, commercial vehicles, marine vehicles and
11 maritime vehicles. Also, composites according to the present
12 invention are readily manufactured, for example, by resin transfer
13 molding (RTM), resin infusion molding, or resin injection molding.

14
15 Having described the invention, the following examples are
16 given to illustrate specific applications of the invention
17 including the best mode now known to perform the invention. These
18 specific examples are not intended to limit the scope of the
19 invention described in this application.

20
21 EXAMPLES

22
23 Example 1: Preparation of phthalonitrile prepolymer

24 Phthalonitrile monomer, 4,4'-bis(3,4-dicyanophenoxy)biphenyl,
25 was purchased from Daychem Laboratories. 10 g of the monomer was

placed in an aluminum planchet and melted on a hot plate at 250°C (monomer melts around 235°C). The monomer melt was degassed for about 2h to eliminate trace amounts of solvent present. The phthalonitrile prepolymer was synthesized by adding 0.15- 0.168 g (1.5-1.68 wt%) of 1,3-bis(3-aminophenoxy)benzene, obtained from National Starch Corporation, to the monomer melt. The melt was stirred for 15 min. and was used for fabrication of phthalonitrile/metal foam composite specimens.

Example 2: Fabrication of phthalonitrile/aluminum foam composite

An aluminum mold, 2"x0.6"x0.2", coated with a teflon mold release agent was used for fabrication of composite specimens. Two aluminum foam strips (1"x0.5"x0.185", density 6-8 % of the solid material, pore size - 40 pores per inch (ppi)) separated by a teflon film, were placed in the mold and heated to 250°C. Approximately 2-3 g of the prepolymer melt synthesized as described above in Example 1 with 1.68 wt% curing additive was poured over the metal foam and degassed for about 15 min. with periodic venting to ensure a good flow of the resin throughout the metal foam. The mold was then heated in an air circulating oven for 9h at 280°C and cooled back to room temperature over a 3h span. The composite samples made with this prepolymer showed an incomplete penetration of the resin into the metal foam. Therefore, subsequent composite fabrications involved a slower curing prepolymer made with 1.5% curing additive. The prepolymer has an initial lower viscosity.

Example 3: Fabrication of phthalonitrile/aluminum foam composite

An aluminum mold, 2"x0.6"x0.2", coated with a teflon mold release agent was used for fabrication of composite specimens. Two aluminum foam strips (1"x0.5"x0.185", density 6-8 % of the solid material, pore size - 40 ppi) separated by a teflon film, were placed in the mold and heated to 250°C. Approximately 2-3 g of the prepolymer melt synthesized with 1.5 wt% curing additive was poured over the metal foam and degassed for about 15 min. with periodic venting to ensure a good flow of the resin throughout the metal foam. The mold was then heated in an air circulating oven for 9h at 280°C and cooled back to room temperature over a 3h span. The composite samples made with this prepolymer showed a complete penetration of the resin through the metal foam. The mechanical and damping properties of the phthalonitrile/aluminum composite samples were evaluated after heat treatment at the following conditions: (A) 9h at 280°C (B) 18h at 280°C (C) 18h at 280, 4h at 325°C and (D) 18h at 280, 4h at 325, 4h at 375°C. Conditions (C) and (D) employed an inert atmosphere purge of argon.

Example 4: Fabrication of phthalonitrile/copper foam composite

An aluminum mold, 2"x0.6"x0.2", coated with a teflon mold release agent was used for fabrication of composite specimens. Two copper foam strips (1"x0.5"x0.185", density 6-8 % of the solid material, pore size - 10 ppi), separated by a teflon film, were placed in the mold and heated to 250°C. The prepolymer melt

1 synthesized as described above in Example 1 with 1.5 wt% curing
2 additive was poured over the metal foam and degassed with periodic
3 venting to ensure a good flow of the resin throughout the metal
4 foam. The mold was then heated in an air circulating oven for 9h
5 at 280°C and cooled back to room temperature over a 3h span. The
6 mechanical and damping properties of the phthalonitrile/copper
7 composite samples were evaluated after heat treatment at the
8 following conditions: (A) 9h at 280°C (B) 18h at 280°C (C) 18h at
9 280, 4h at 325°C and (D) 18h at 280, 4h at 325, 4h at 375°C.
10 Conditions (C) and (D) employed an inert atmosphere purge of argon.

11
12 Example 5: Fabrication of phthalonitrile/titanium foam composite

13 An aluminum mold (2"x0.6"x0.2", density 6-8 % of the solid
14 material, pore size - 10 ppi) coated with a teflon mold release
15 agent was used for fabrication of composite specimens. Two
16 titanium foam strips, 1"x0.5"x0.185"x0.185", separated by a teflon
17 film, were placed in the mold and heated to 250°C. The prepolymer
18 melt synthesized as described above in Example 1 was poured over
19 the metal foam and degassed for about 15 min. with periodic venting
20 to ensure a good flow of the resin throughout the metal foam. The
21 mold was then heated in an air circulating oven for 9h at 280° C
22 and cooled back to room temperature over a 3h span. The mechanical
23 and damping properties of the phthalonitrile/titanium composite
24 samples were evaluated after heat treatment at the following
25 conditions: (A) 9h at 280°C (B) 18h at 280°C (C) 18h at 280, 4h at

325°C and (D) 18h at 280, 4h at 325, 4h at 375°C. Conditions (C) and (D) employed an inert atmosphere purge of argon.

Example 6: Fabrication of phthalonitrile/zinc foam composite

An aluminum (mold, 2"x0.6"x0.2", density 6-8 % of the solid material, pore size - 10 ppi) coated with a teflon mold release agent was used for fabrication of composite specimens. Two zinc foam strips, 1"x0.5"x0.185", separated by a teflon film, were placed in the mold and heated to 250°C. The prepolymer melt synthesized as described above in Example 1 with 1.5% curing additive was poured over the metal foam and degassed for about 15 min. with periodic venting to ensure a good flow of the resin throughout the metal foam. The mold was then heated in an air circulating oven for 9h at 280°C and cooled back to room temperature over a 3h span. The mechanical and damping properties of the phthalonitrile/zinc composite samples were evaluated after heat treatment at the following conditions : (A) 9h at 280°C (B) 18h at 280°C (C) 18h at 280, 4h at 325°C and (D) 18h at 280, 4h at 325, 4h at 375°C. Conditions (C) and (D) employed an inert atmosphere purge of argon.

Example 7. Fabrication of Aluminum Alloy Foam / Rubber (White) Composite.

An aluminum mold, 2"x0.6"x0.2", coated with a teflon mold release agent was used for fabrication of composite specimens. Two

1 aluminum foam strips, 1"x0.5"x0.185", separated by a teflon film,
2 were placed in the mold. The aluminum foam strips were submerged in
3 the polymeric uncured natural rubber dissolved in an organic
4 solvent and with a small amount of peroxide to thermally cure the
5 rubber. The composite was subjected to pressure to remove the
6 solvent and to consolidate the composite fabrication. The
7 impregnated uncured rubber/aluminum foam composition was heated at
8 about 160°C for 1-2 hours to crosslink the rubber.

9
10 Example 8. Fabrication of Aluminum Alloy Foam /Red Rubber
11 Composite.

12 An aluminum mold, 2"x0.6"x0.2", coated with a teflon mold
13 release agent was used for fabrication of composite specimens. Low
14 viscosity uncured RTV silicone rubber was quickly placed in the
15 mold and two aluminum foam strips, 1"x0.5"x0.185", separated by a
16 teflon film, were submerged in the uncured rubber. The mold was
17 then placed in a vacuum oven at reduced pressure for 15 minutes.
18 The rubber was then cured at room temperature for 24 hours
19 affording a rubber/aluminum foam composite.

20
21 Example 9. Fabrication of Epoxy/Aluminum Alloy Foam Composite.

22 An aluminum mold, 2"x0.6"x0.2", coated with a teflon mold
23 release agent was used for fabrication of composite specimens. Epon
24 828 and an aromatic diamine were mixed thoroughly and placed into
25 the aluminum mold. Several aluminum foam strips, 1"x0.5"x0.185",

1 separated by a teflon film, were submerged in the epoxy/amine
2 composition at approximately 100°C in a vacuum oven. At this time,
3 a vacuum was applied to consolidate the dispersion of the
4 composition into the pores of the aluminum foam. The composition
5 was cured by heating at 65°C for 5-6 hours. The epoxy/aluminum foam
6 composite was used for evaluating damping and mechanical
7 properties.

8
9 Example 10. Fabrication of Acrylic/Aluminum Alloy Foam Composite.

10 An aluminum mold, 3"x 2" was used for fabrication of composite
11 specimens. 18 ml of catalyst B was added to 40 ml of resin A of
12 the EPO-KWICK components and thoroughly mixed. The mixture was
13 poured on top of several aluminum foam strips, 1"x0.5"x0.185",
14 contained in the aluminum mold. At this time, a vacuum was applied
15 for 10 minutes to degas and consolidate the dispersion of the
16 composition into the pores of the aluminum foam. The composition
17 was cured by keeping at room temperature overnight. The
18 acrylic/aluminum alloy foam composite was used for evaluating
19 damping and mechanical properties.

20
21 Example 11. Preparation of Porous Metal/Organic Polymeric Composite
22 Samples for Damping Measurements.

23 For the purpose of demonstration, we have used selected foams
24 and filler materials. Many metals or even high strength alloys can
25 be used as skeleton material and also wide variety of filler

1 materials can be used to design the composite possessing required
2 strength and damping capability. Other factors such as
3 environmental compatibility, temperature and chemical compatibility
4 in addition to cost can dictate the choice of the materials to be
5 used.

6
7 The composites produced in Examples 3-9 were used for damping
8 measurements. The samples were prepared with dimensions having 4.5
9 mm thickness, 10 mm width, and 32 mm length. The damping capacity
10 of the samples were measured with a dynamic mechanical thermal
11 analyzer (DMTA). A small sinusoidal mechanical stress is applied to
12 the sample and the resulting sinusoidal strain transduced.
13 Comparison of the amplitude of the signals yields the complex
14 dynamic modulus E^* . The phase lag (δ) of strain behind stress is
15 measured and the storage modulus and loss factor of the material
16 are calculated.

17
18 Example 12. Damping Characteristics of Aluminum Alloy
19 Foam/Phthalonitrile Composite.

20 The aluminum alloy foam/phthalonitrile composite prepared in
21 Example 3 was evaluated for its damping characteristics under 1 Hz
22 frequency. The results of the damping measurements were shown in
23 Fig. 1. The figure shows the damping behavior of the composite
24 under different heat treatment conditions as described in Example
25 3. The damping measurements are plotted with temperature. It is

1 noted that damping peak locations at a given temperature can be
2 adjusted based on heat treatment of composite. The room temperature
3 damping (flat portion of curve) is also higher than the best
4 damping material such as Vacrosil™ as shown in Fig. 10.

5
6 Example 13. Damping Characteristics of Copper Foam/Phthalonitrile
7 Composite.

8 The copper foam/phthalonitrile composite prepared in Example
9 4 was evaluated for its damping characteristics under 0.1 to 10 Hz
10 frequency. The results of the damping measurements were shown in
11 Fig. 2, Fig. 3, and Fig. 4. The figures show the damping behavior
12 of the composite under different heat treatment conditions as
13 described in Example 4. The damping measurements are plotted with
14 temperature. It is noted that damping peak locations at a given
15 temperature can be adjusted based on heat treatment of the
16 composite. The room temperature damping (flat portion of curve) is
17 also higher than the best damping material such as Vacrosil™ as
18 shown in Fig. 10.

19
20 Example 14. Damping Characteristics of Titanium Foam/Phthalonitrile
21 Composite.

22 The titanium foam/phthalonitrile composite prepared in Example
23 5 was evaluated for its damping characteristics under 0.1 to 10 Hz
24 frequency. The results of the damping measurements were shown in
25 Fig. 5, Fig. 6, and Fig 7. The figures show the damping behavior of

1 the composite under different heat treatment conditions as
2 described in Example 5. The damping measurements are plotted with
3 temperature. It is noted that damping peak locations at a given
4 temperature can be adjusted based on heat treatment of the
5 composite. The room temperature damping (flat portion of curve) is
6 also higher than the best damping material such as Vacrosil™ as
7 shown in Fig. 10.

8
9 Example 15. Damping Characteristics of Zinc Foam/Phthalonitrile
10 Composite.

11 The zinc foam/phthalonitrile composite prepared in Example 6
12 was evaluated for its damping characteristics under 0.1 to 10 Hz
13 frequency. The results of the damping measurements were shown in
14 Fig. 8 and Fig. 9. The figures show the damping behavior of the
15 composite under different heat treatment conditions as described in
16 Example 5. The damping measurements are plotted with temperature.
17 It is noted that damping peak locations at a given temperature can
18 be adjusted based on heat treatment of the composite. The room
19 temperature damping (flat portion of curve) is also higher than the
20 best damping material such as Vacrosil™ as shown in Fig. 10.

21
22 Example 16. Damping Characteristics of Aluminum Alloy Foam/white
23 rubber Composite.

24 The aluminum alloy foam/white composite prepared in Example 7
25 was evaluated for its damping characteristics under 0.1 to 10 Hz

1 frequency. The results of the damping measurements were shown in
2 Fig. 11. This figure shows the damping behavior of the composite.
3 The damping measurements are plotted with temperature. It is noted
4 that damping characteristics are better than the best damping
5 material such as Vacrosil™ as shown in Fig. 10.

6
7 Example 17. Damping Characteristics of Aluminum Alloy Foam/red
8 rubber Composite.

9 The Aluminum Alloy Foam/red rubber composite prepared in
10 Example 8 was evaluated for its damping characteristics under 0.1
11 to 10 Hz frequency. The results of the damping measurements were
12 shown in Fig. 12. This figure shows the damping behavior of the
13 composite. The damping measurements are plotted with temperature.
14 It is noted that damping characteristics are better than the best
15 damping material such as Vacrosil™ as shown in Fig. 10.

16
17 Example 18. Damping Characteristics of Aluminum Alloy Foam/Epoxy
18 Composite.

19 The Aluminum Alloy Foam/epoxy composite prepared in Example 8
20 was evaluated for its damping characteristics under 0.1 to 10 Hz
21 frequency. The results of the damping measurements were shown in
22 Fig. 13. This figure shows the damping behavior of the composite.
23 The damping measurements are plotted with temperature. It is noted
24 that damping characteristics are better than the best damping
25 material such as Vacrosil™ as shown in Fig. 10.

1 Example 19. Damping Characteristics of Aluminum Alloy Foam/Acrylic
2 Composite.

3 The Aluminum Alloy Foam/Acrylic composite prepared in Example
4 10 was evaluated for its damping characteristics under 0.1 to 10 Hz
5 frequency. The results of the damping measurements were shown in
6 Fig. 14. The figure shows the damping behavior of the composite.
7 The damping measurements are plotted with temperature. It is noted
8 that damping characteristics are better than the best damping
9 material such as Vacrosil™ as shown in Fig. 10.

10
11 Example 20. Mechanical Properties of Aluminum Alloy
12 Foam/Phthalonitrile Composite.

13 The aluminum alloy foam/phthalonitrile composite prepared in
14 Example 3 was evaluated for its room temperature compressive
15 mechanical properties after various heat treatment exposures. For
16 example, Fig. 15 shows the stress/strain diagram for the composite
17 which was heat treated at 280°C for 18 hours, at 325°C for 4 hours,
18 and at 375°C for 4 hours. The figure shows that the composite
19 exhibit superior mechanical properties relative to the cured
20 phthalonitrile resin.

21
22 Example 21. Mechanical Properties of Aluminum Foam/Acrylic
23 Composite.

24 The aluminum alloy foam/Acrylic composite prepared in Example
25 10 was evaluated for its room temperature compressive mechanical

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1 properties after various heat treatment exposures. For example,
2 Fig. 16 shows the stress/strain diagram for the composite. The
3 figure shows that the composite exhibit superior mechanical
4 properties relative to the aluminum alloy foam.

5
6 Additional information concerning the present invention may be
7 found in the copending United States Patent Application of Imam,
8 Sastri and Keller, entitled LIGHTWEIGHT HIGH DAMPING POROUS
9 METAL/PHTHALONITRILE COMPOSITES, filed on even date herewith, the
10 entirety of which is incorporated herein by reference.

11
12 Obviously, many modifications and variations of the present
13 invention are possible in light of the above teachings. It is
14 therefore to be understood that, within the scope of the appended
15 claims, the invention may be practiced otherwise than as
16 specifically described.

Claims

What is claimed is:

1. An acoustically damping composite article, comprising a metal foam, said metal foam having an open cell structure, impregnated with a solid, bulk non-elastomeric polymer.
2. The composite article of claim 1, wherein said metal is selected from the group consisting of aluminum, aluminum base alloys, titanium, titanium base alloys, nickel, nickel base alloys, copper, copper base alloys, iron, iron base alloys, zinc, zinc base alloys, lead, lead base alloys, silver, silver base alloys, gold, gold base alloys, platinum, platinum base alloys, tantalum, and tantalum base alloys.
3. The composite article of claim 1, wherein said polymer is selected from the groups consisting of epoxies, acrylics, hardened silicones, polyurethanes, polyimides, polyvinyls, polycarbonates, hardened natural rubbers, hardened synthetic rubbers, phenolics, polyolefins, polyamides, polyesters, fluoropolymers, poly(phenylene ether ketones), poly(phenylene ether sulfones), poly(phenylene sulfides) and melamine-formaldehyde resins.

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4. The composite article of claim 1, wherein said metal is an aluminum base alloy foam.

5. The composite article of claim 1, wherein said metal is a copper foam or a copper base alloy foam.

6. The composite article of claim 1, wherein said metal is a zinc foam or a zinc base alloy foam.

7. The composite article of claim 3, wherein said metal is an aluminum foam or an aluminum base alloy foam.

8. The composite article of claim 3, wherein said metal is a copper foam or a copper base alloy foam.

9. The composite article of claim 3, wherein said metal is a zinc foam or a zinc base alloy foam.

10. The composite article of claim 3, wherein said metal is a titanium foam or a titanium base alloy foam.

11. The composite article of claim 1, wherein said polymer is an epoxy.

12. The composite article of claim 1, wherein said polymer is an

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acrylic.

13. The composite article of claim 1, wherein said polymer is a hardened silicone rubber.

14. The composite article of claim 1, wherein said polymer is a hardened natural rubber.

15. The composite article of claim 1, wherein said polymer is a hardened synthetic non-silicone rubber.

16. The composite article of claim 1, wherein said polymer is a phenolic.

17. The composite article of claim 1, wherein said cells have a locally uniform diameter.

18. The composite article of claim 17, wherein said metal foam has a gradation of pores sizes in at least one direction along the metal foam.

19. A composite article according to claim 1, wherein said composite article is in the form of a sheet.

20. A laminate comprising a plurality of sheets according to claim

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19 bonded together.

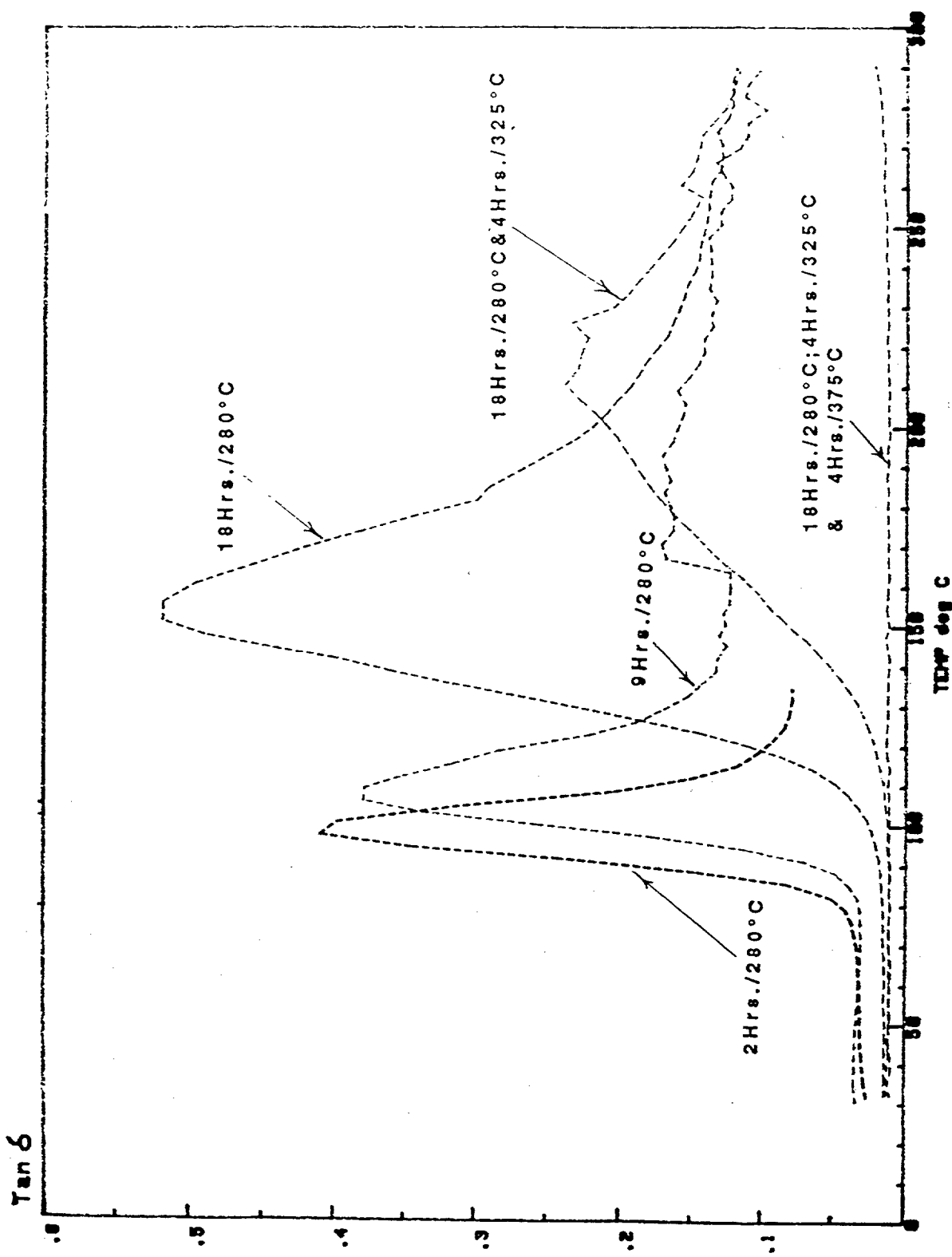
21. An acoustically damping composite article, comprising a metal foam, said metal foam having an open cell structure, impregnated with solid bulk polymer, said metal foam being, along its smallest dimensions, no smaller than 3 times the average diameter of said cells..


22. A method of forming a composite comprising the steps of:
impregnating a metal foam, said metal foam having an open cell structure, with a resin component; and
converting said resin component, within said cells, to a bulk solid, non-elastomeric polymerized resin.

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268240" 26854800





PL
DMTA

1 Hz

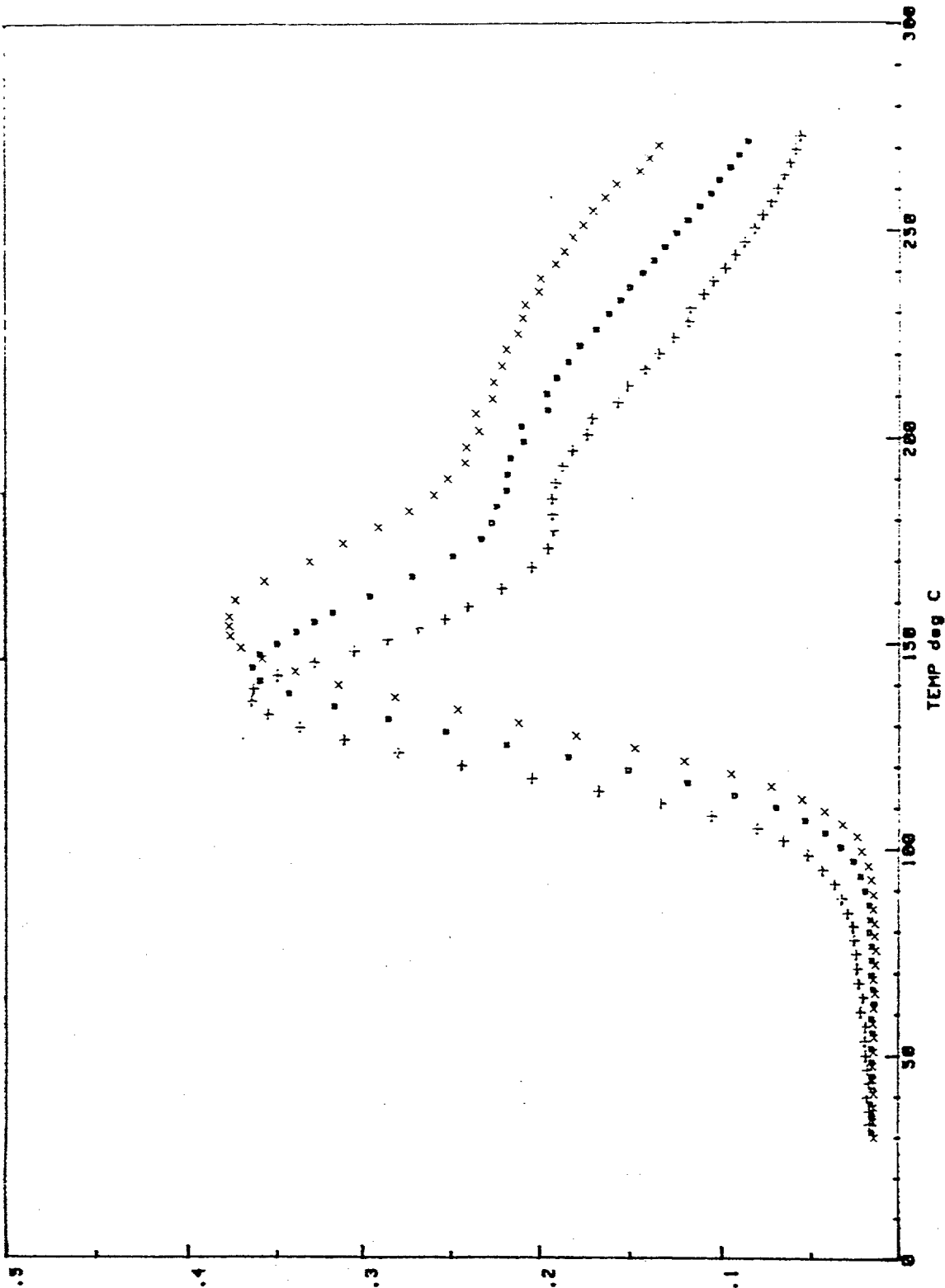
0.1, 1 & 10
STRAIN max 4
2 degC/min
-LOCK = 2.510
DUAL CANY
4.588300552222222

Fig 1

268497880

Cu-Ph
9H0280C

Tan δ



DMTA

x 10 Hz
square 1 Hz
+ 0.1 Hz

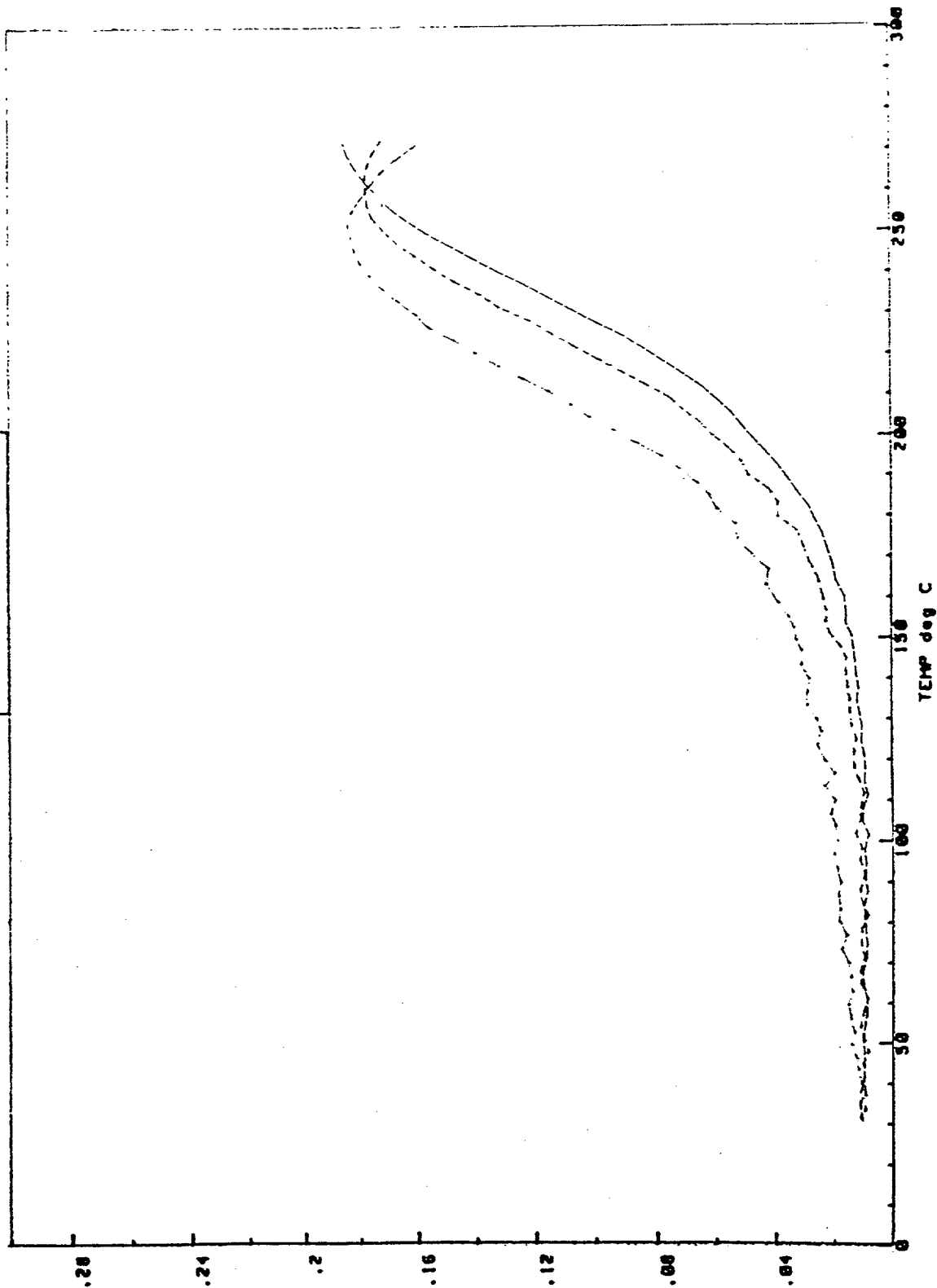
0.1, 1 & 10
STRAIN = 0.4
2 degC/min
-LOGE = 2.82
DUAL CANT
4.45x9.96x7mm

Fig. 2

26340" 263430

Cu-Ph
16H0280C;4H0325C

Tan δ



DMTA

--- 10 Hz
--- 1 Hz
... 0.1 Hz

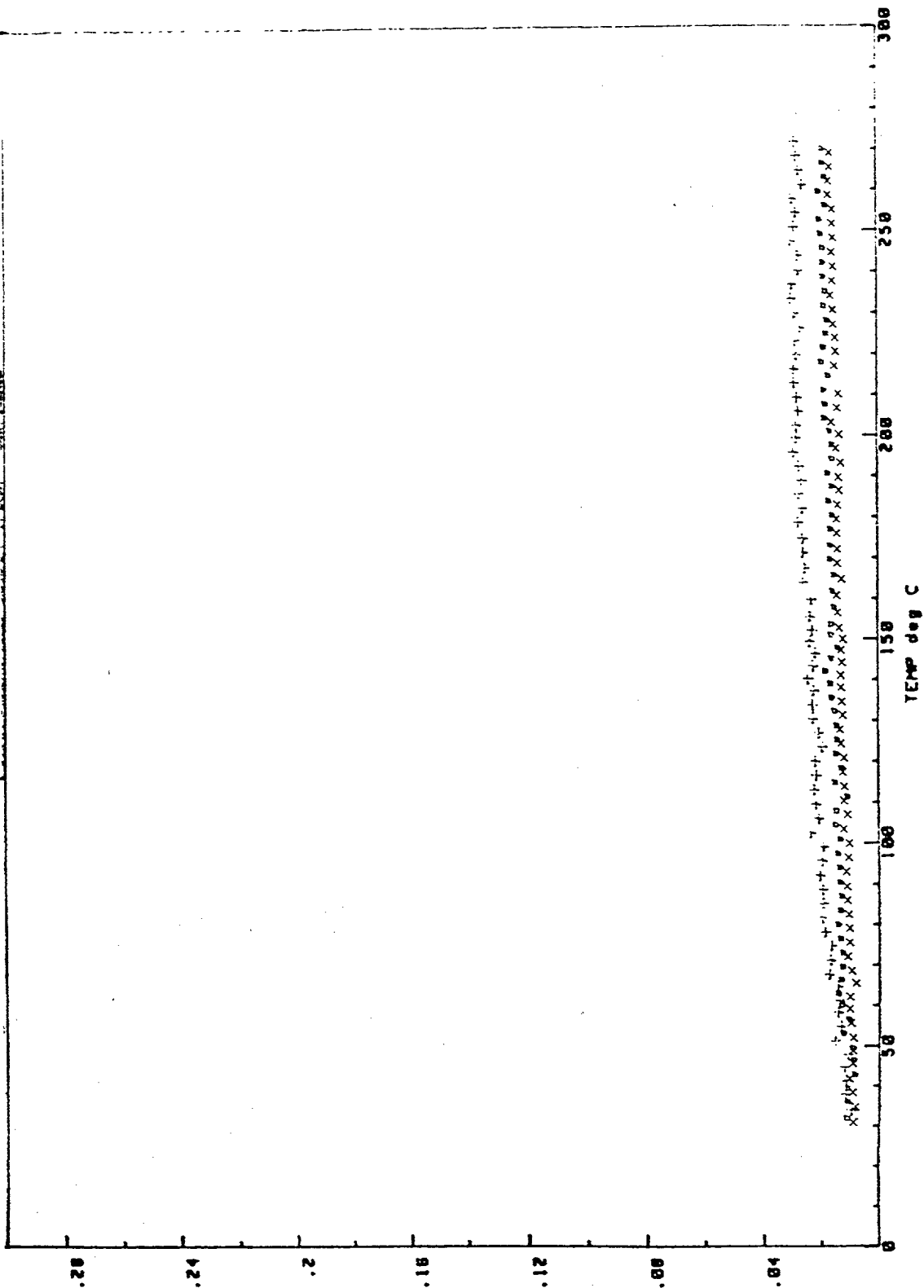
0.1, 1 & 10
STRAIN = 4
2 degC/min
-LOG₁₀ = 2.682
DUAL CANT
4.21x3.79x7mm

Fig. 3

468240" 26854830

Cu-Ph
16H0280C;4H0325C;4H0375C

Tan δ



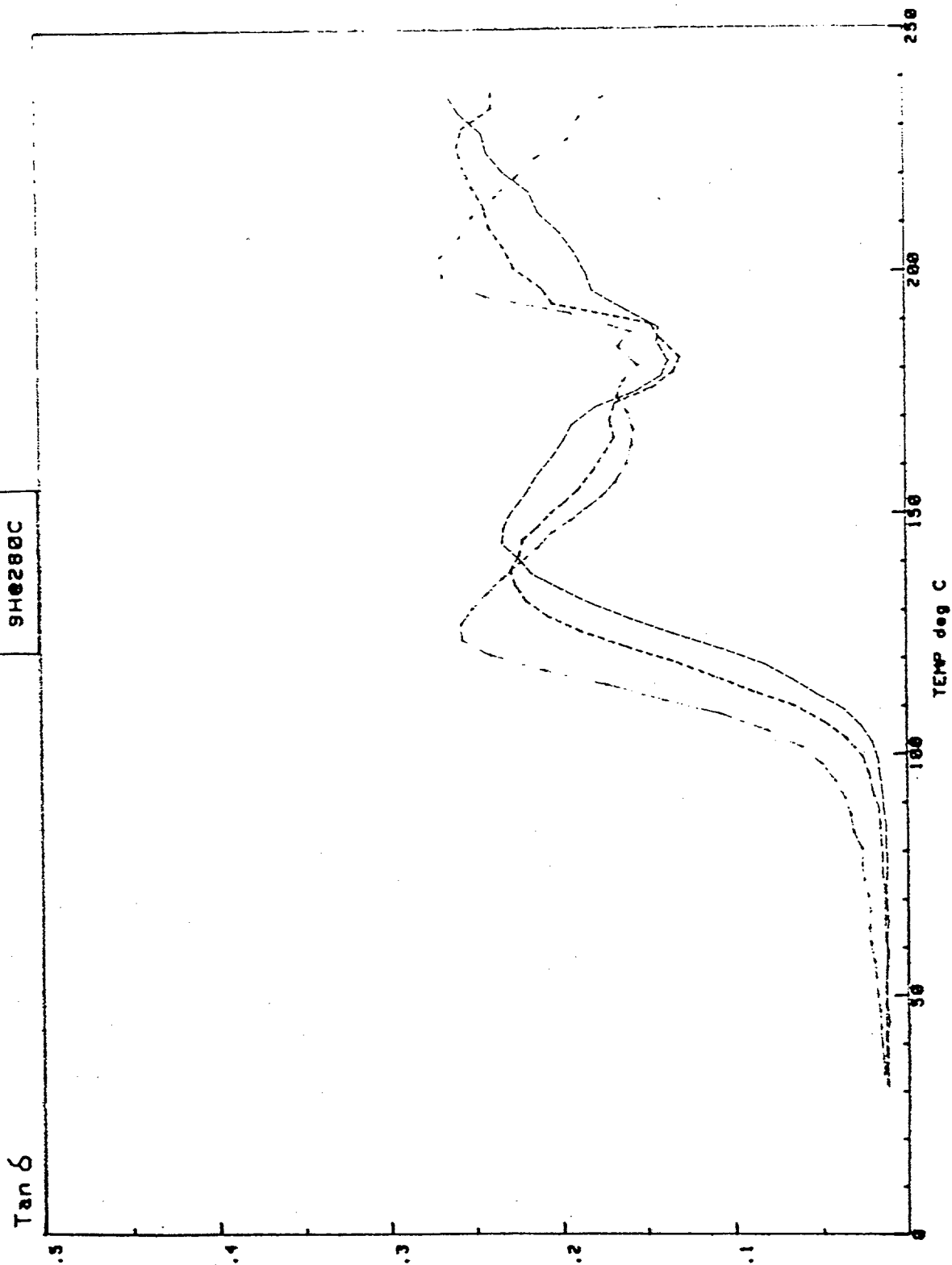
DMTA

x 10 Hz
□ 1 Hz
+ .1 Hz

0.1, 1 & 10
STRAIN = .04
2 degC/min
-LOCK = 2.583
DUAL CANT
4.21x9.77x7mm

Fig. 4

268340-2684880



PL DMTA

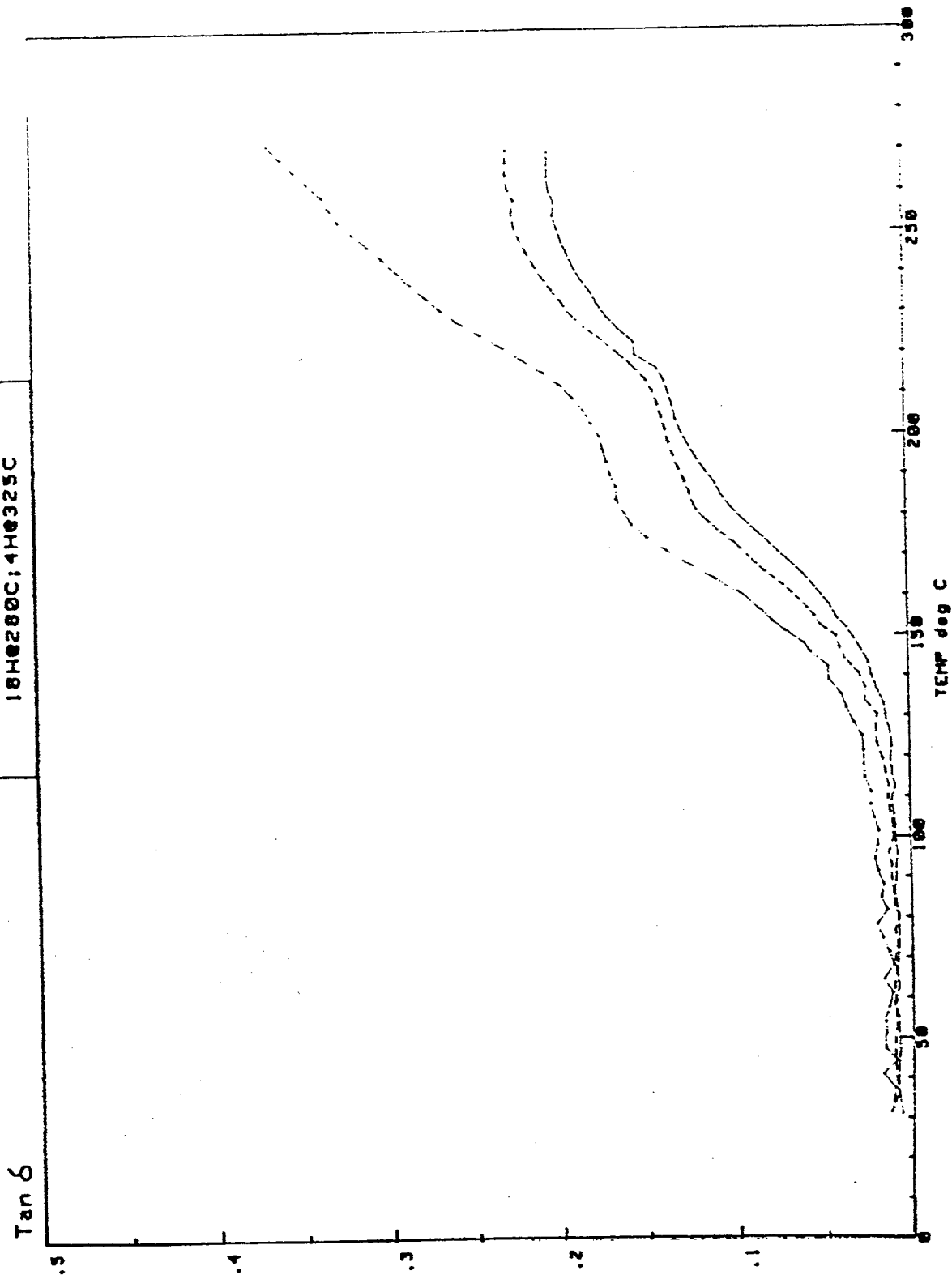
--- 10 Hz
... 1 Hz
- . - . 0.1 Hz

0.1, 1 & 10
STRAIN = 4
2 degC/min
- LOCK = 2.689
DUAL CANT
4.54x10.81x7mm

Fig. 5

463240" 46354380

Ph/Ti Composite
18H0280C;4H0325C



PL DMTA

--- 10 Hz
... 1 Hz
- 0.1 Hz

0.1, 1 & 10
STRAIN = 0.4
2 degC/min
-LOGK = 2.615
DUAL CANT
4.51x10x7mm

Fig. 6

263240" 26354330

Ph/Ti Composite
18H0280C;4H0325C;4H0375C

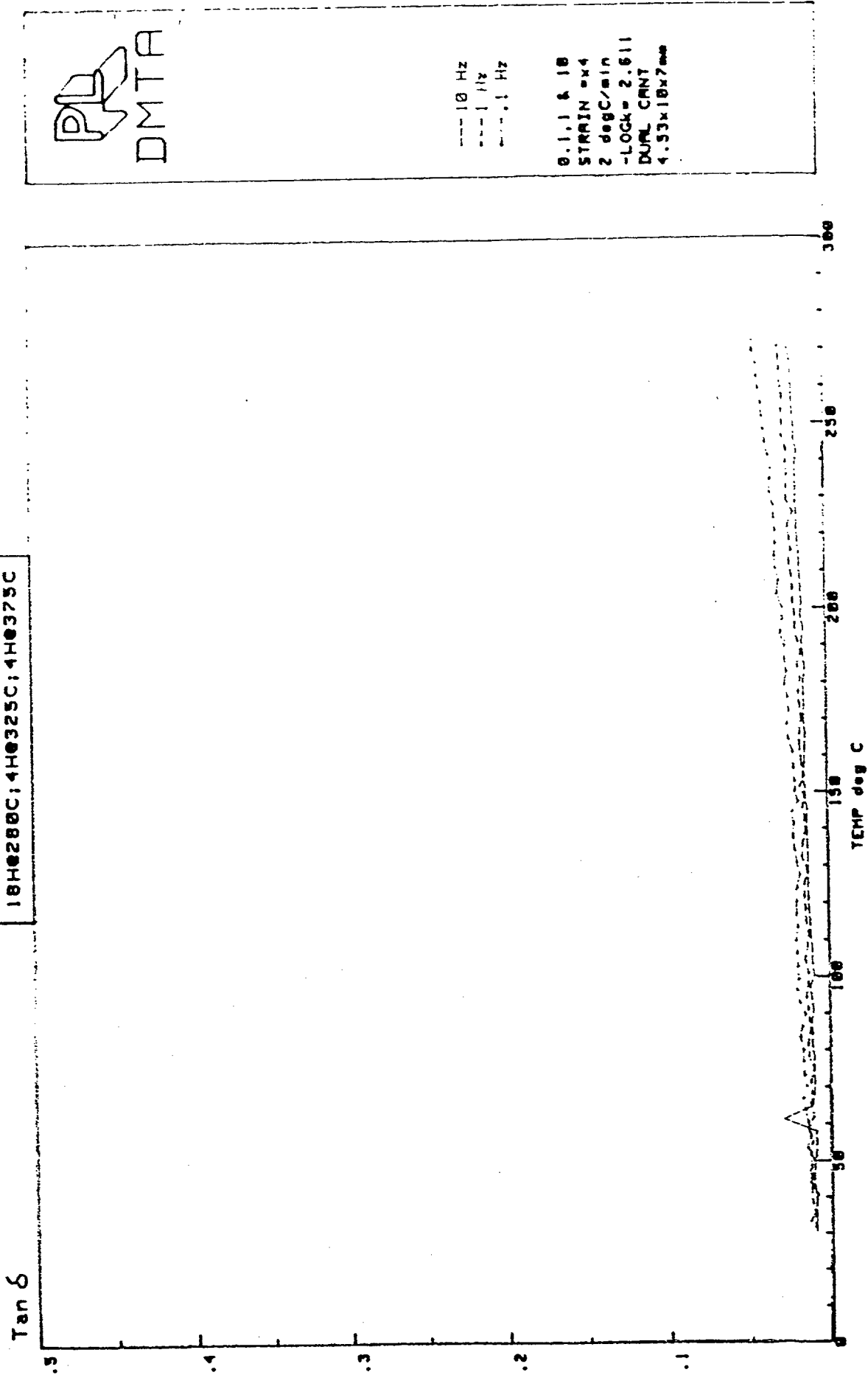
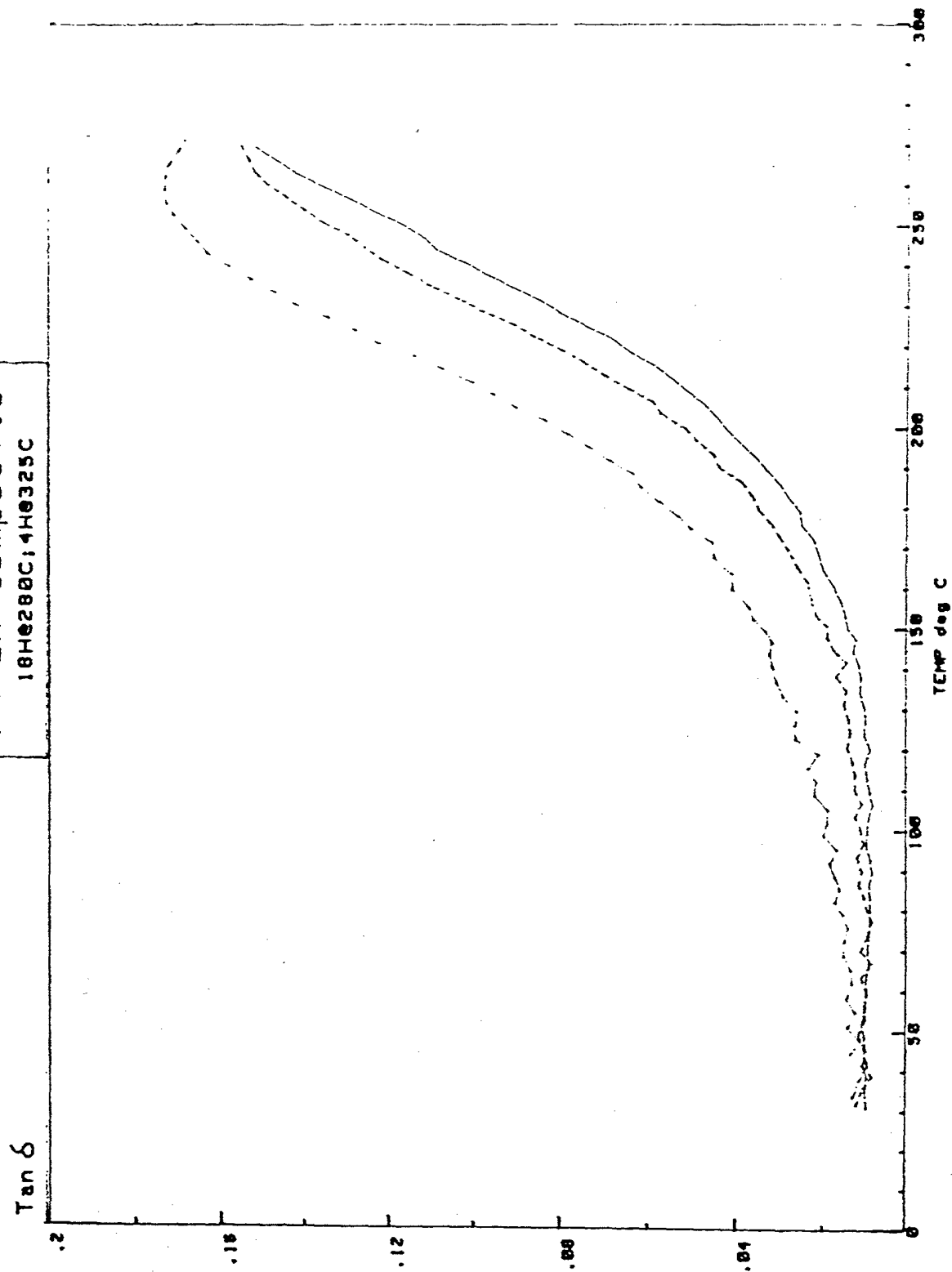


Fig. 7

468240" 46854880

Ph/Zn Composite
18H0280C, 4H0325C



PL
DMTA

--- 10 Hz
- - - 1 Hz
... 0.1 Hz
8.1, 1 & 10
STRAIN = x4
2 degC/min
-LOGK = 2.615
DUAL CANT
4.51x10x7mm

Fig. 8

268240" 26854830

Ph/Zn Composite
18H0280C;4H0325C;4H0375C

Tan δ

.2
.16
.12
.08
.04
0

TEMP deg C
50 100 150 200 250 300

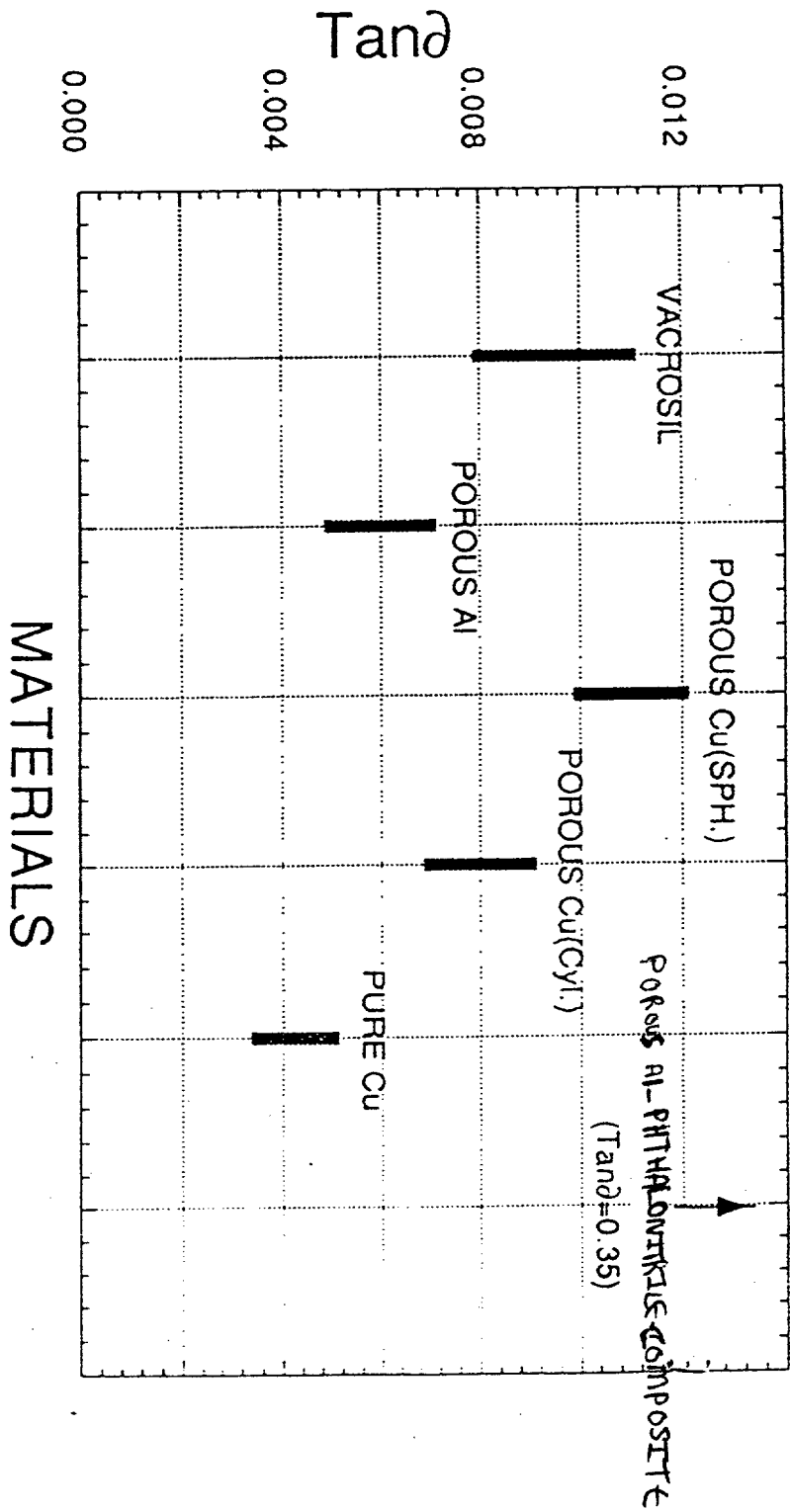


--- 10 Hz
- - - 1 Hz
_ _ _ .1 Hz

0.1, 1 & 10
STRAIN = .4
2 degC/min
-LOCK = 2.615
DUAL CANT
4.51x10x7mm

Fig. 9

Tan δ Over Frequency Range of 0.1 to 10 Hz



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268240 25854880

WHITE RUBBER AL FORM

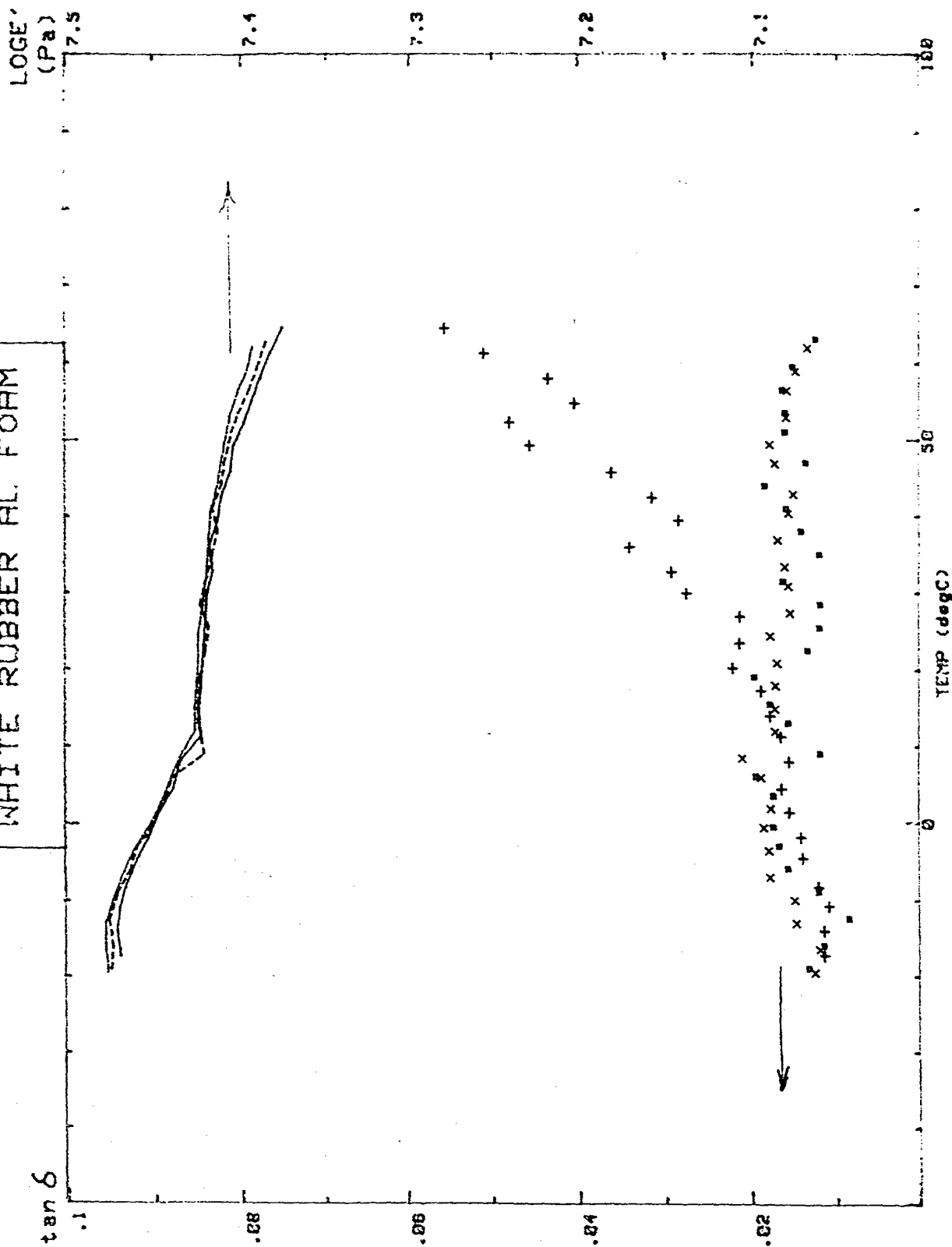
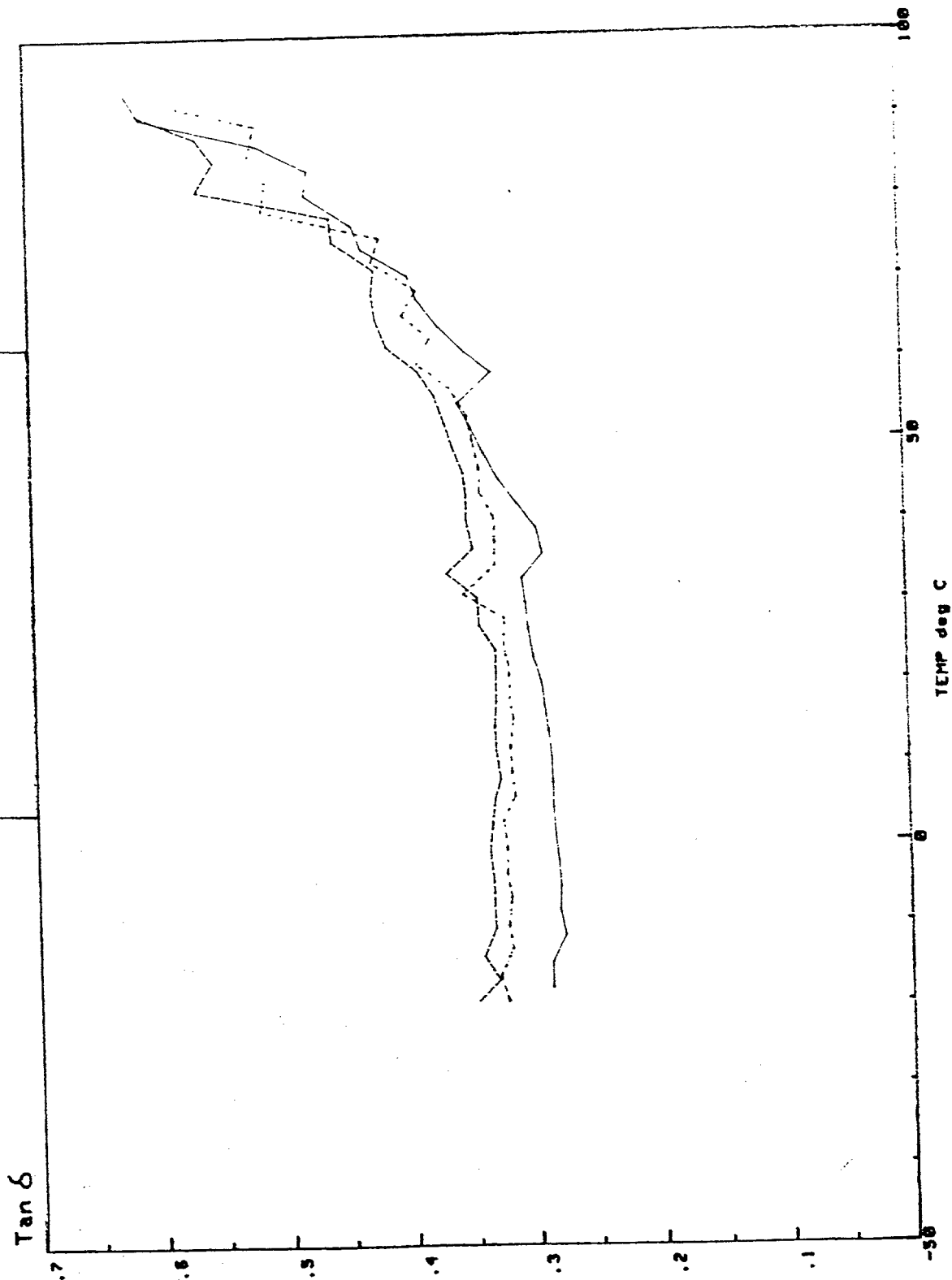


Fig. 11

263410" 26354880

RED RUBBER AL FORM



PL
DMTA

--- 10 Hz

---- 1 Hz

0.1, 1 & 10

STRAIN = 4

2 degC/min

-LOGA = 3.863

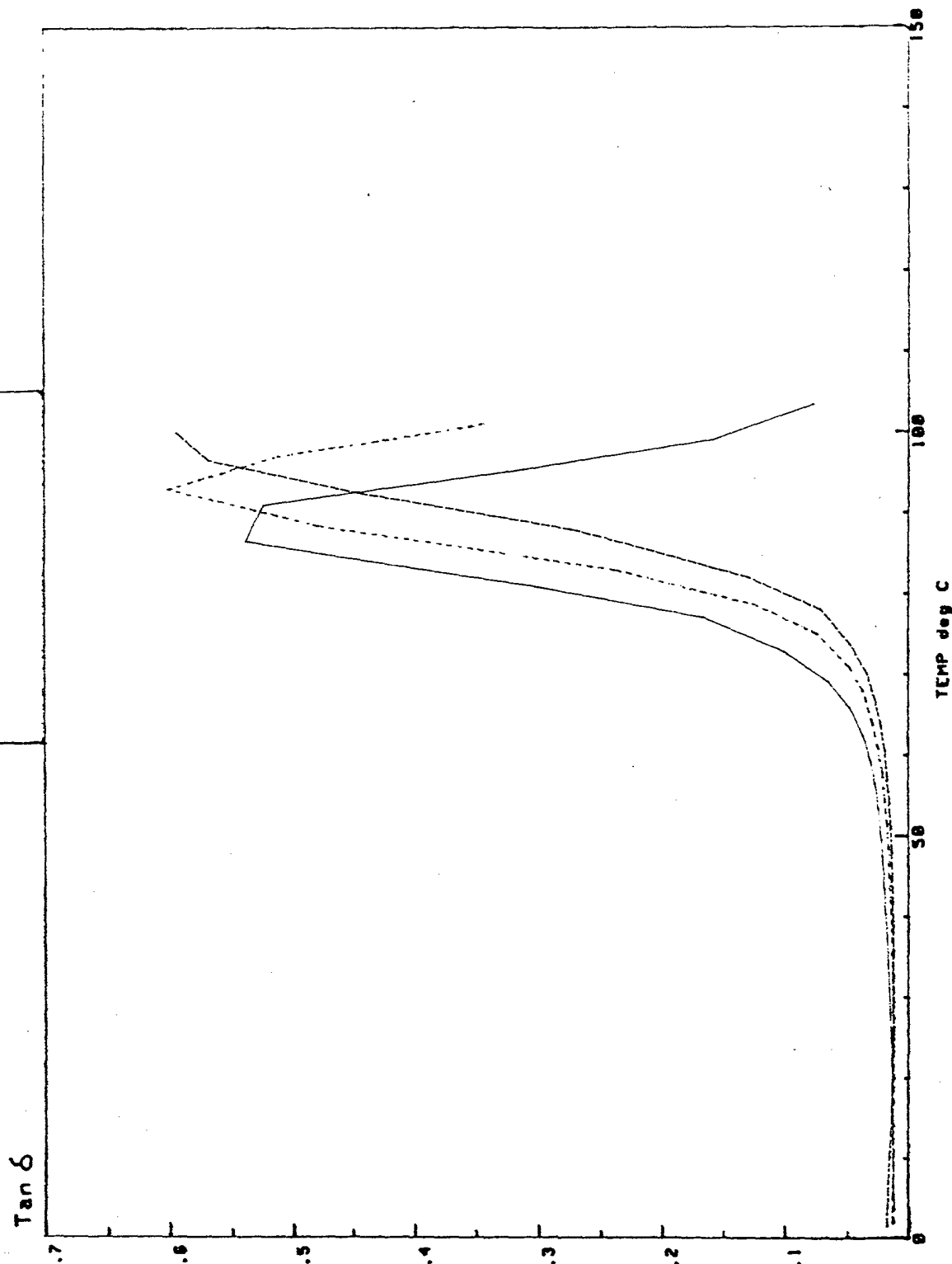
SINGLE CANT

4.6x11.3x13mm

Fig. 12

268240" 26854880

EPOXY AL FORM



DMTA

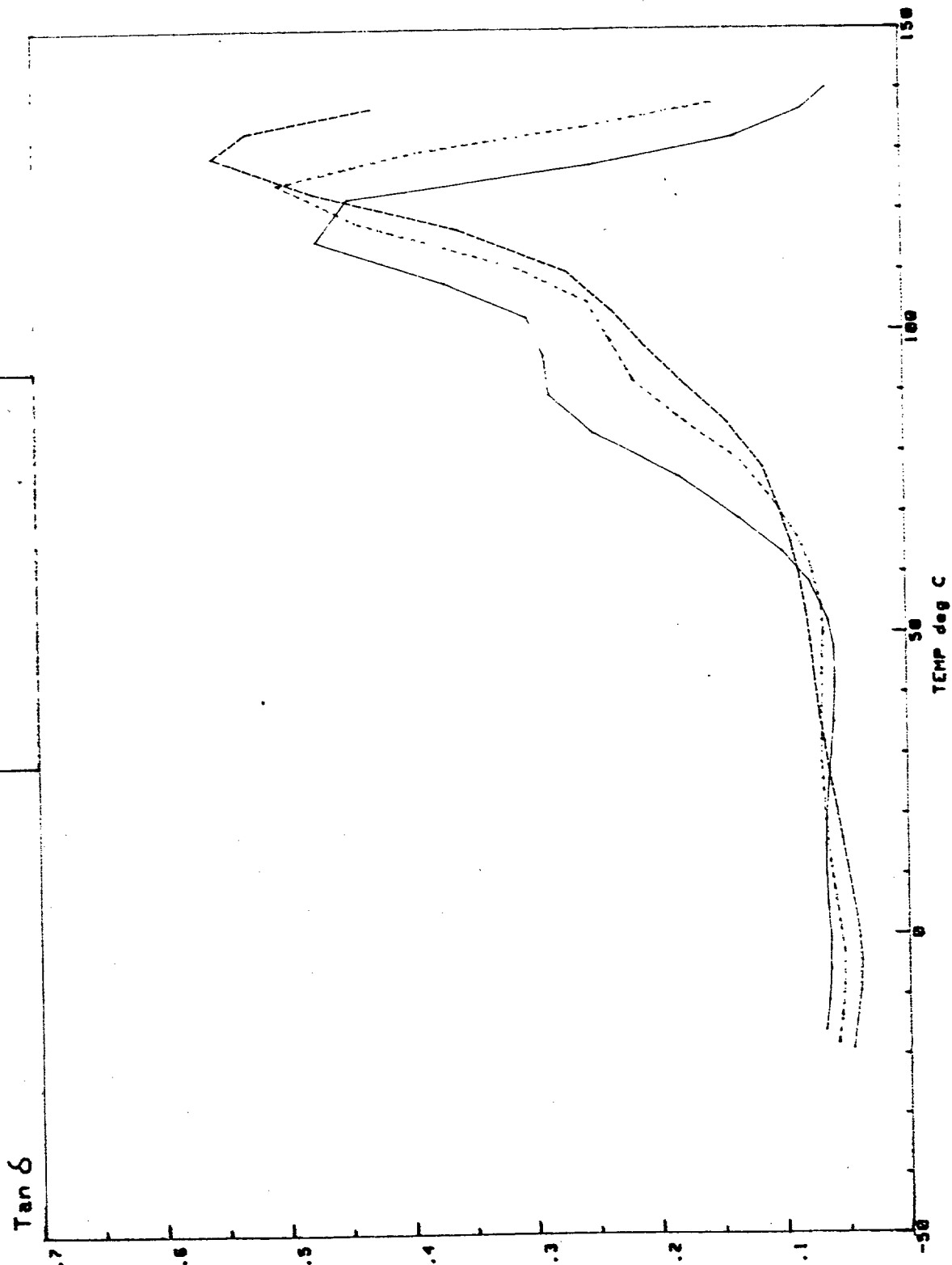
--- 10 Hz
- - - 1 Hz
- - - 0.1 Hz

0.1, 1 & 10
STRAIN = 0.4
2 degC/min
-LOGk = 3.978
SINGLE CANT
4.31x10.33x19mm

Fig. 13

268340" 26854830

ACRYLIC AL FORM



PL DMTA

10 Hz

1 Hz

0.1 Hz

0.1, 1 & 10

STRAIN = 4

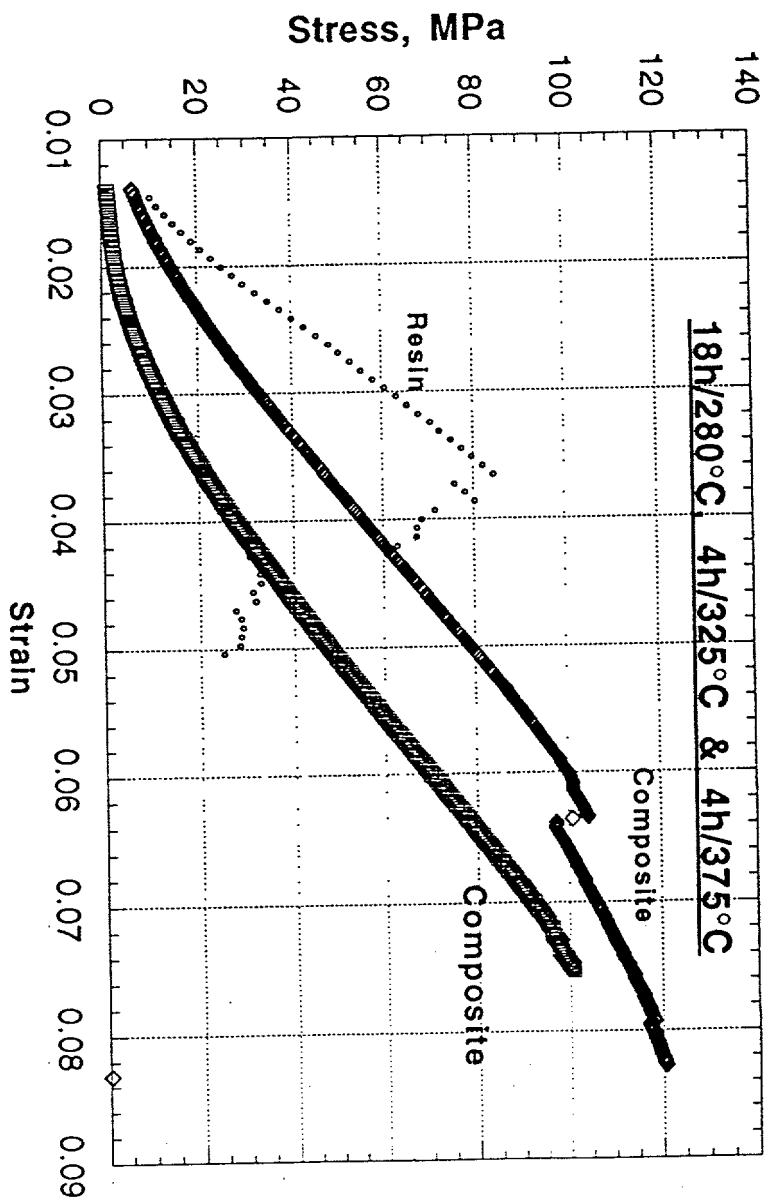
3 degC/min

-LOGK = 4.895

SINGLE CANT

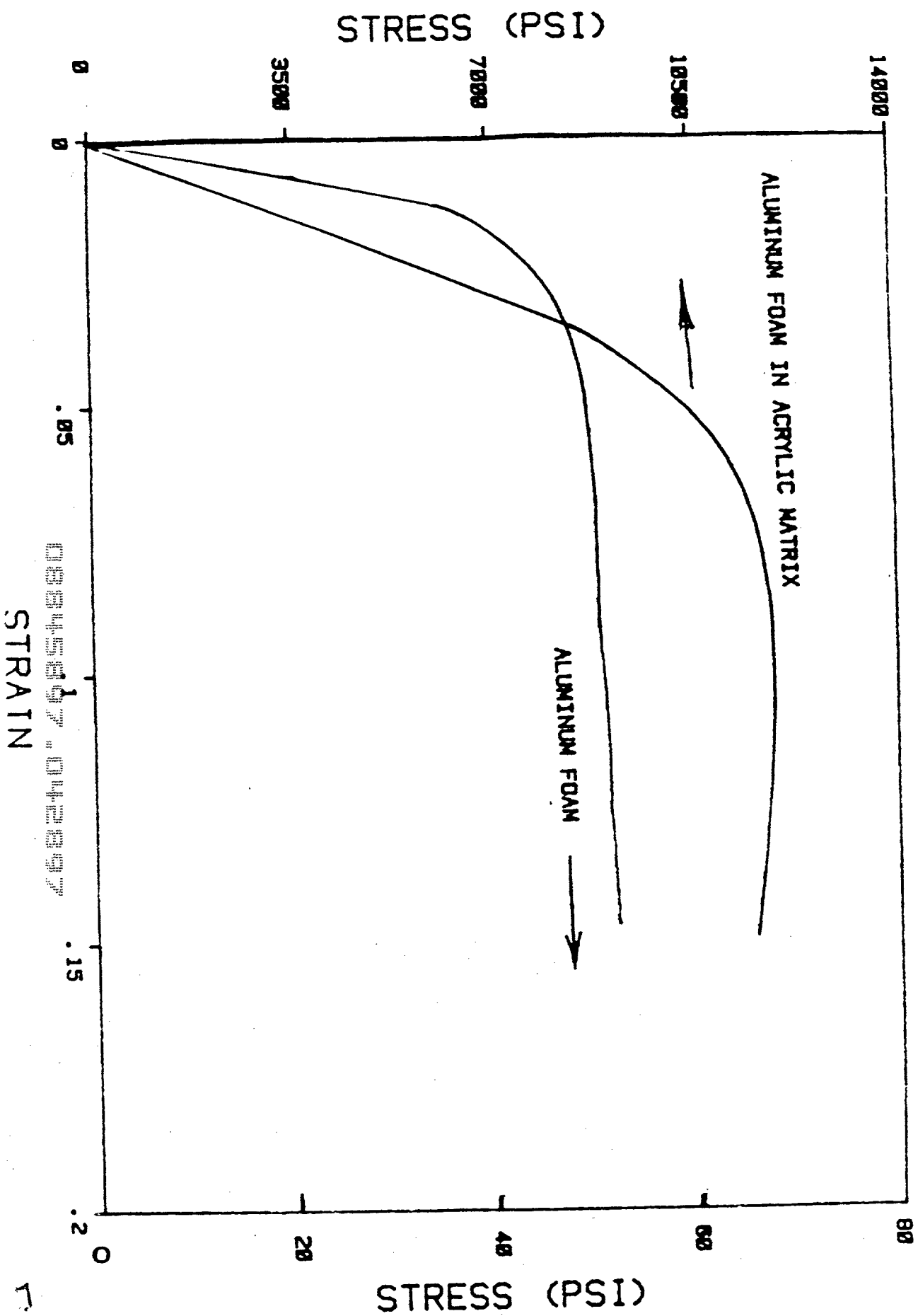
3.834x10.94x19mm

Fig. 14



03845897 042897

STRESS VS STRAIN



As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

(check one) ☒ is attached hereto. ☐ was filed on _____ as
Application Serial No. _____
and was amended on _____

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

Prior Foreign Applications			Priority Claimed	
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
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Inventor's signature B. D. Calk Date 4/28/97

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Citizenship: USA

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Declaration and Power of Attorney For Patent Application

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled: "POROUS METAL/ORGANIC POLYMERIC COMPOSITES" the specification of which:

(check one) ☒ is attached hereto. ☐ was filed on _____ as
Application Serial No. _____
and was amended on _____.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign applications for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Applications			Priority Claimed	
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No

I hereby claim the benefit under Title 35, United States Code, §120 of any United States applications listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status)
		(patented, pending, abandoned)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorneys/and/or agent/s/ to prosecute this application and transact all business in the Patent and Trademark Office connected therewith, and hereby certify that the Government of the United States has the irrevocable right to prosecute this application:

Thomas E. McDonnell,
Reg., No. 26950

Barry A. Edelberg
Reg. No. 31012

SEND CORRESPONDENCE TO:
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Washington, D.C. 20375-5000

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(202)-404-1558

I hereby declare that all statements made herein of own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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